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TECHNOLOGY CENTER

ARF Project D217

Contract No. DA-18-108-405-CML-777

INHIBITION OF FLASHING OF AEROSOLS

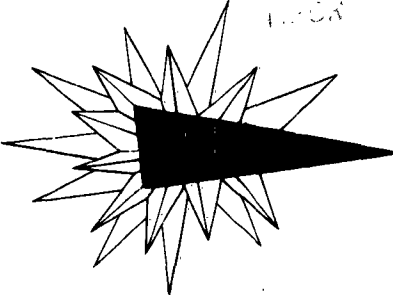
Quarterly Progress Report V

C. C. Miesse

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25 years of research



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ARMOUR RESEARCH FOUNDATION OF
ILLINOIS INSTITUTE OF TECHNOLOGY

DA-18-108-405-CML-777

Quarterly Progress Report No. V

April 15, 1961 to July 14, 1961

INHIBITION OF FLASHING OF AEROSOLS

C. C. Miesse

July 15, 1961

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ARMOUR RESEARCH FOUNDATION OF ILLINOIS INSTITUTE OF TECHNOLOGY

The fifth Quarterly Progress Report summarizes the work performed during the past quarter, from April 15, 1961 to July 14, 1961, under Contract No. DA-18-108-405-CML-777, on the study of "Inhibition of Flashing of Aerosols", for the Physical Chemistry Division of the Army Chemical Center, Edgewood, Maryland.

The following personnel have contributed to this project during the fifth quarter: C. C. Miesse and D. K. Werle.

Data are recorded in Armour Research Foundation Logbook C-10313.

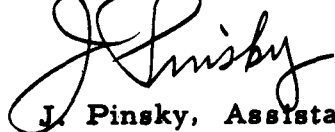
Respectfully submitted,

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C. C. Miesse, Principal Investigator

APPROVED:



J. Pinsky, Assistant Director of
Fluid Dynamics and Propulsion Research

CCM/aa

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INHIBITION OF FLASHING OF AEROSOLS

I. ABSTRACT

This report covers the period between April 15, 1961 to July 14, 1961, during which a number of revealing quantitative and qualitative tests were performed with dibutyl phthalate (DBP) and bis (2-ethylhexyl) hydrogen phosphite (B2EHHP) aerosols. Both aerosols were found to be quite flammable, and the B2EHHP aerosol flame front, on one occasion, propagated several times faster than the DBP during its tests. DBP lean limits of 27.8 - to 17.3 mg/l for 74 - to 130 micron mean weight diameter aerosols indicate that the lean limit decreases as the drop size increases in this size range.

Ignition studies indicated that it was not possible to ignite flammable concentrations of DBP aerosols with spark discharge or exploded wire ignition systems. Hot wire, 110V shorted nichrome wire, and gas flame ignition systems resulted in identical lean limits in DBP, although the gas flame ignition system is the most positive ignition source.

II. INTRODUCTION

This project under Contract DA-18-108-405-CML-777 was initiated on April 15, 1960, by the Army Chemical Center, to investigate fundamental behavior in the flashing of flammable liquid aerosols so that means of prevention or inhibition of the flashing may be revealed. The variables to be investigated include mass concentration, drop size,

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liquid volatility, ignition source, and, if possible, pressure and temperature. In the second phase of this study, flame inhibiting additives will be investigated.

During the fifth quarter the experimental apparatus was modified to permit more accurate control of experimental conditions, and a better quantitative determination of the resultant data. Drop size distributions of the DBP aerosol were determined by the magnesium-coated slide technique, and concentrations were measured gravimetrically. Of the several ignition sources investigated, the 1.5 cm gas flame proved to be the most reliable for ignition of both DBP and Bis (2-ethylhexyl) hydrogen phosphite. Flammable concentration limits for the former were found to decrease with the Sauter Mean Diameter of the aerosol.

III. MODIFIED EXPERIMENTAL APPARATUS

In this report period, the combustion test equipment was transferred from the Fluids Laboratory to the Chemistry Laboratory, in order to provide adequate ventilation facilities for removal of toxic fumes. A ten-foot-high plastic hood enclosure (Fig. 1) was built around the equipment, and was connected to an existing hood ventilation duct. Since many of the inhibitors that will be used in this study are toxic or have toxic decomposition products, these changes were necessary to minimize danger to the operating personnel.

A. Thermal Aerosol Generator and Combustion Tube

Some modifications were made in the test equipment as shown in Figs. 2 and 3. The gate valve immediately below the condenser was removed and the nitrogen-air exhaust port was widened, to reduce turbulence.

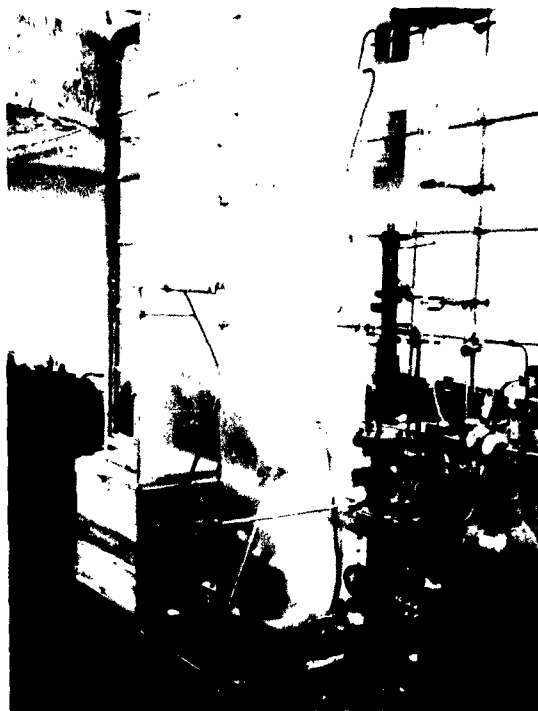


Fig. 1 **THE COMBUSTION TEST FACILITY**

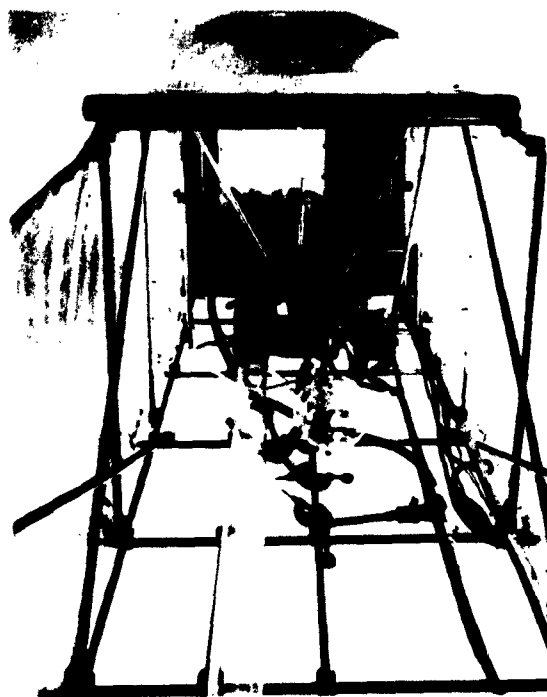


Fig. 2 **THE THERMAL AEROSOL GENERATOR**

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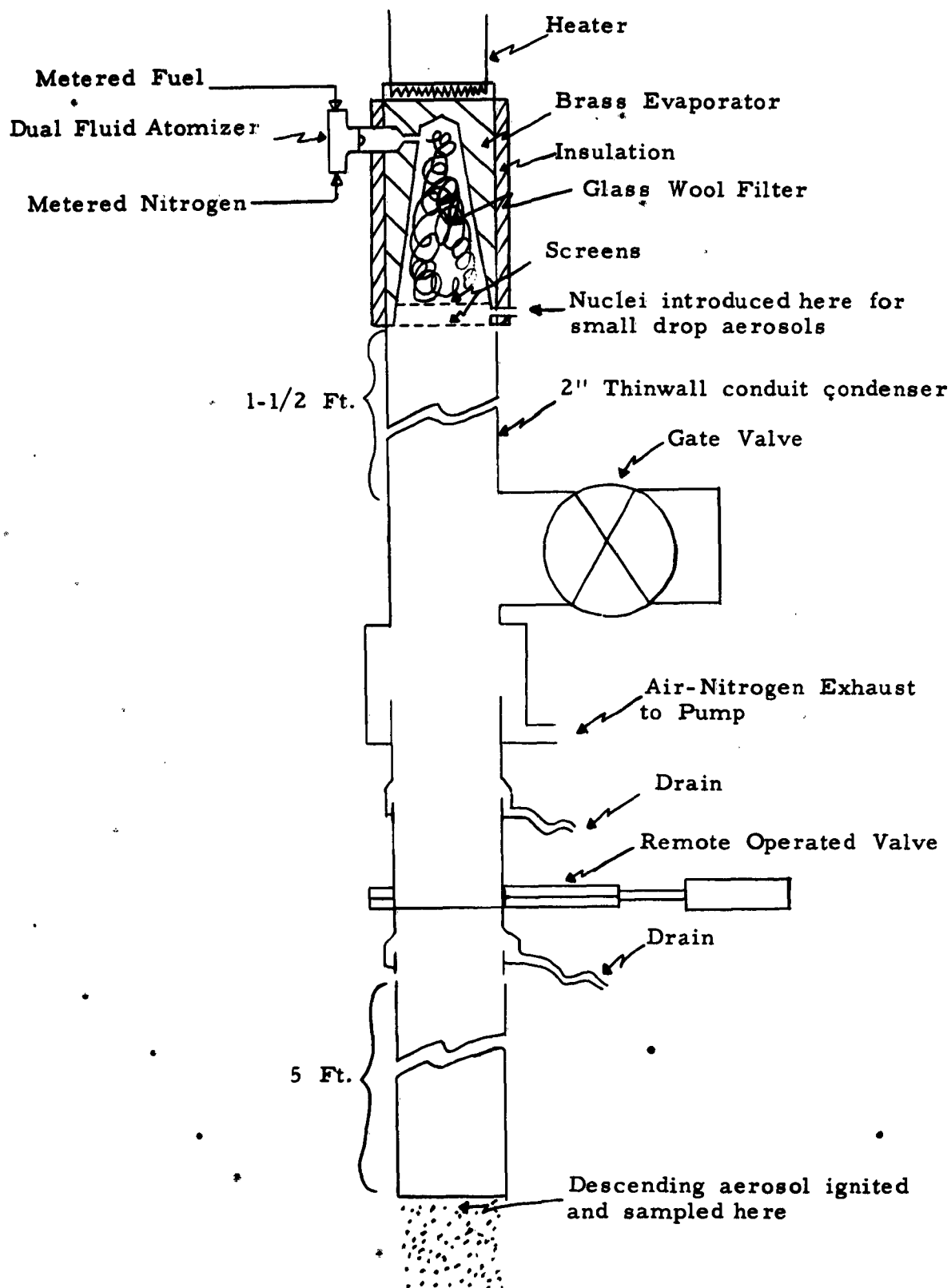


Fig. 3 SCHEMATIC DIAGRAM OF MODIFIED COMBUSTION APPARATUS

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A rotameter was placed in the nitrogen line so that the nitrogen flow could be measured directly. The steel fuel reservoir was replaced with a six-inch diameter glass pipe, to facilitate the changing of fuels and/or additives. Also, a remote-operated two-inch full port slide valve was designed and built, and mounted above the combustion tube, so that the entire aerosol in the volume of the combustion tube could be isolated from the aerosol generator and sampled directly. In the tests performed in the previous report periods, the mass concentration was determined by dividing the mass flow rate of liquid by the terminal velocity of the drops, as calculated from the Sauter Mean Diameter of the aerosol. Any errors in the measurement of the aerosol drop size distribution would then result in an erroneous mass concentration (mg/l). Use of the remote-operated valve permitted direct measurement of the mass of aerosol per unit volume, and eliminated the inherent errors in the former method of indirect measurement.

B. Large Drop Aerosol Generator

The large drop aerosol generator shown in Fig. 4 is currently under construction, and is awaiting delivery of a commercial remote-operated ball valve for completion. A Bete P-20 spray nozzle, operated at about 10 psig, will produce a relatively uniform DBP aerosol in the 400 - to 500 micron size range when the spray is elutriated to eliminate the smaller drops. With a rising air column, only the large drops with a settling velocity greater than the rising air velocity will descend into the combustion tube. Since earlier work indicated difficulty in attaining flammable elutriated concentrations with a single spray nozzle, provisions are being made for a multiple spray system which will provide a higher mass

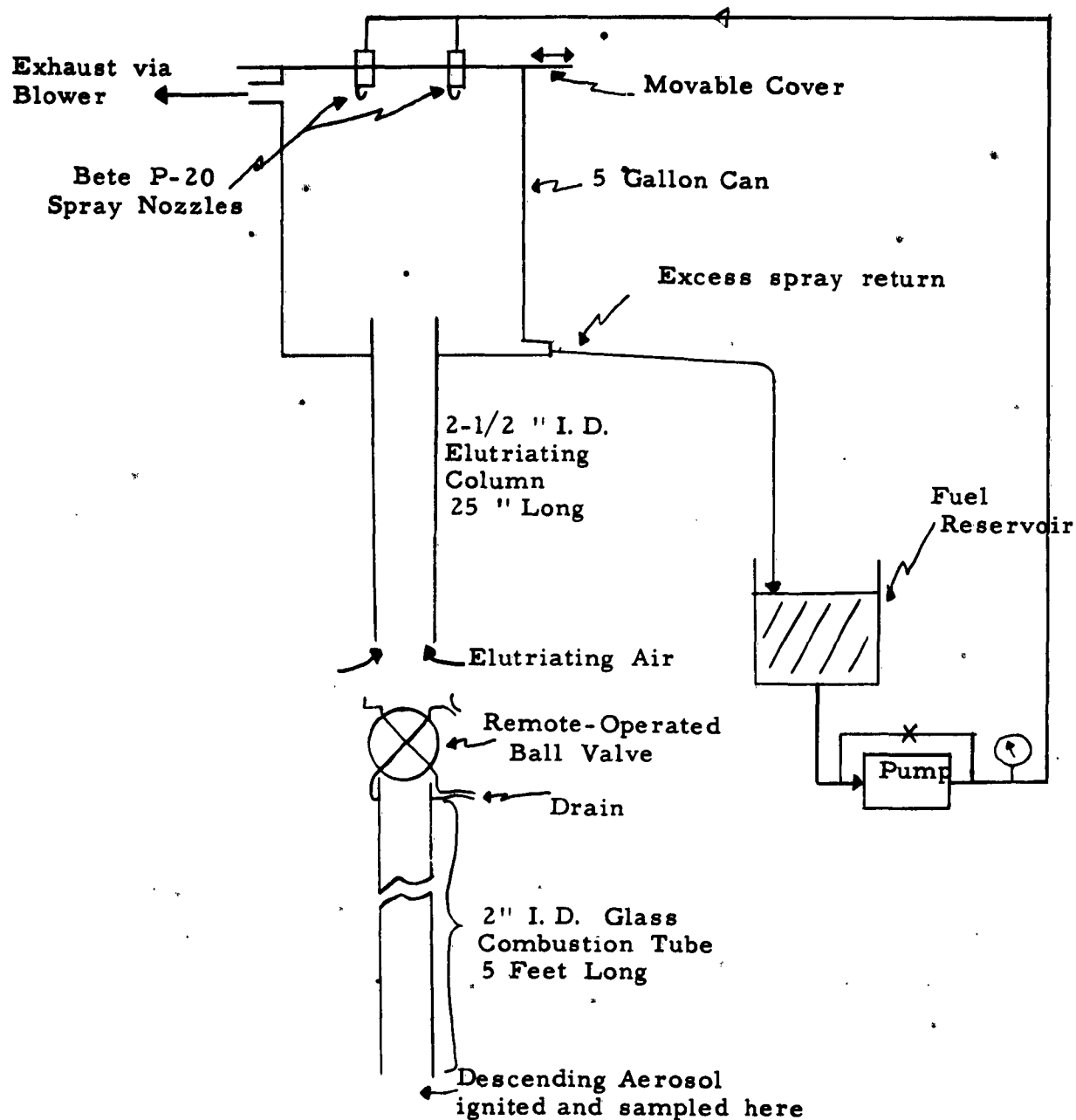


Fig. 4 LARGE DROP AEROSOL GENERATOR

concentration. The relatively higher liquid flow rate required by these spray nozzles will be supplied by a peripheral turbine pump that will provide recirculation of the fuel which does not descend through the elutriating column.

C. Ignition Techniques

Semi-quantitative tests were conducted to determine the feasibility of ignition with spark discharge, exploded wires, and hot wires. A high voltage (10 KV) capacitor bank (26 - to 52 μ f) was utilized to determine spark and exploding wire ignition capabilities for 100 - to 500 micron diameter dibutyl phthalate aerosols as produced by a large drop aerosol generator, consisting of a Bete P-20 spray nozzle contained in a five-gallon container. The ignition source was placed at the bottom of a 2-1/2 inch ID glass tube, which was attached to the aerosol generator. In no case was it possible to ignite the DBP aerosol with spark energies ranging from 102 - to 786 joules. Although A. E. Weller (Ref. 1) reported that ignition of a kerosene aerosol required 160 millijoules for an ignition frequency of 25 per cent, and 4 joules for an ignition frequency of 100 per cent, the low volatility of DBP accounts for its non-ignitability by spark discharge. It may be possible to improve ignitability by lengthening the spark duration, however. Ignition with exploded wires was also attempted (16.6 - to 468 joules), but ignition invariably failed. In each case, following ignition failure by both spark discharge and exploded wire, the aerosol was easily ignited with a 1-1/2 cm long gas flame. Thus the aerosol concentration was within flammable limits for all tests. Since a 7/8 inch long 30 gauge Nichrome wire shorted across 110 V usually ignited the aerosol, the longer duration of the 110 V discharge apparently accounts

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for the difference in ignition susceptibility.

Hot wire ignition proved successful for temperatures of 1016°C or higher. A B and S 18 gauge platinum wire heated to this temperature barely ignited the DBP aerosol - after a 5 to 10 second ignition lag. At higher temperatures ignition occurred more readily, and at 1300°C ignition took place as soon as the wire was introduced into the aerosol. Wire temperatures were measured with an optical pyrometer (Leeds and Northrup). Finer platinum wires were examined (24, 30 and 34 gauge) and the minimum ignition temperature appeared to rise slightly with decreasing wire diameter, probably due to the thermal instability of the finer wires (temperature varied markedly with air movement).

The possible catalytic effect of platinum as a hot wire ignition source was investigated by substituting a pure iron wire. However, the minimum ignition temperature for the DBP aerosol was but 10°C higher with the iron wire and is considered within experimental error.

In the determination of the lean flammable limits, it was found that the same limit was obtained with the hot platinum wire, the 110 Volt shorted nichrome wire, and a 1-1/2 cm gas flame was more dependable and resulted in fewer aborted ignitions than did the hot and shorted wires. Therefore, in tests with an open combustion tube, the 1-1/2 cm gas flame will be used for ignition. When it becomes necessary to run closed system combustion tests (as with gaseous inhibitors), the shorted nichrome wire will be used for ignition. The 1-1/2 cm long gas flame appears to ignite the DBP aerosol in less than one second. One second of the gas flow rate of 1.43 cc/sec for the 1000 BTU/cubic feet gas is equivalent to about 50 joules of energy, considerably less than the energy range covered by the spark and exploded

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wire discharge.

The possible effects of the combustion products of the gas ignitor must be considered. Certainly, the concentration of the combustion products in the immediate vicinity above the ignitor is high. However, the less than 5 cc of combustion products produced in one second mix rapidly with the surrounding air. When one considers that the total volume of the combustion tube is 3360 cc, it does not appear likely that the small volume of combustion products from the ignitor can measurably affect the observed lean limits, especially in view of the fact that the same lean limit was obtained with the hot platinum wire and the shorted nichrome wire.

IV. DROP SIZE DISTRIBUTIONS FOR DBP

Drop size data reported in the previous quarterly reports were determined by direct photography of the falling aerosol, a technique which could not be perfected with the limited time and funds available. Because of unsatisfactory photographic definition, and the time-consuming nature of the photographic method, the magnesium oxide coated slide method described by May (Ref. 2) was selected for use in tests described in this report. Burgoyne and Cohen (Ref. 3) used this method for sampling descending tetralin aerosols in the 7 - to 55 micron range. However, the greater settling velocity of the larger drops results in a bias toward the larger drops in direct proportion to the settling velocity. Ideally, representative sampling of the aerosol would require that the entire aerosol within a closed volume be allowed to settle out on a slide. Although the slide valve at the top of the combustion tube could be closed at the same time that a magnesium oxide coated plate was placed at the bottom of the tube, such a sample would be much too dense for sizing purposes. For a countable concentration, a

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sampled height of the order of 2 inches is required; however the high settling velocities of the aerosols under study make such a sampling device impractical. For example, a 150 micron drop will fall 18 inches in one second, or nearly 2 inches in 0.1 sec, thus requiring that the 2 inch high sampled volume be closed off in about 0.01 sec in order to obtain a representative and uniform sample. Of course, the drops larger than 150 microns are even more difficult to sample in this manner.

The above considerations led to the use of a settling correction factor which assumes that during a fixed sampling period (ordinarily about one second or less), drops are sampled from a height or volume which is proportional to the respective settling velocities. By dividing each frequency count of a size range by the respective settling velocity of that size, the inherent bias in sampling a falling aerosol is minimized, if not eliminated entirely. In all of the size distributions presented in this report, the observed size-counts were corrected in this manner.

Some typical frequency variations are shown in Fig. 5. While these are not monodisperse aerosols in the strict sense of the word, the size spread is narrow for such unstable large drop aerosols. As presented in Table I, the arithmetic mean diameter varied from 62 to 119 microns, the mean weight diameter varied from 74 - to 130 microns, the Sauter Mean Diameter varied from 87 - to 147 microns, the weight mean diameter varied from 109 - to 181 microns, and the mass median diameter from 93 to 150 microns. Since the data on hand are insufficient to indicate which mean diameter will correlate best with the flammable concentration, we will continue to report the size data in this manner.

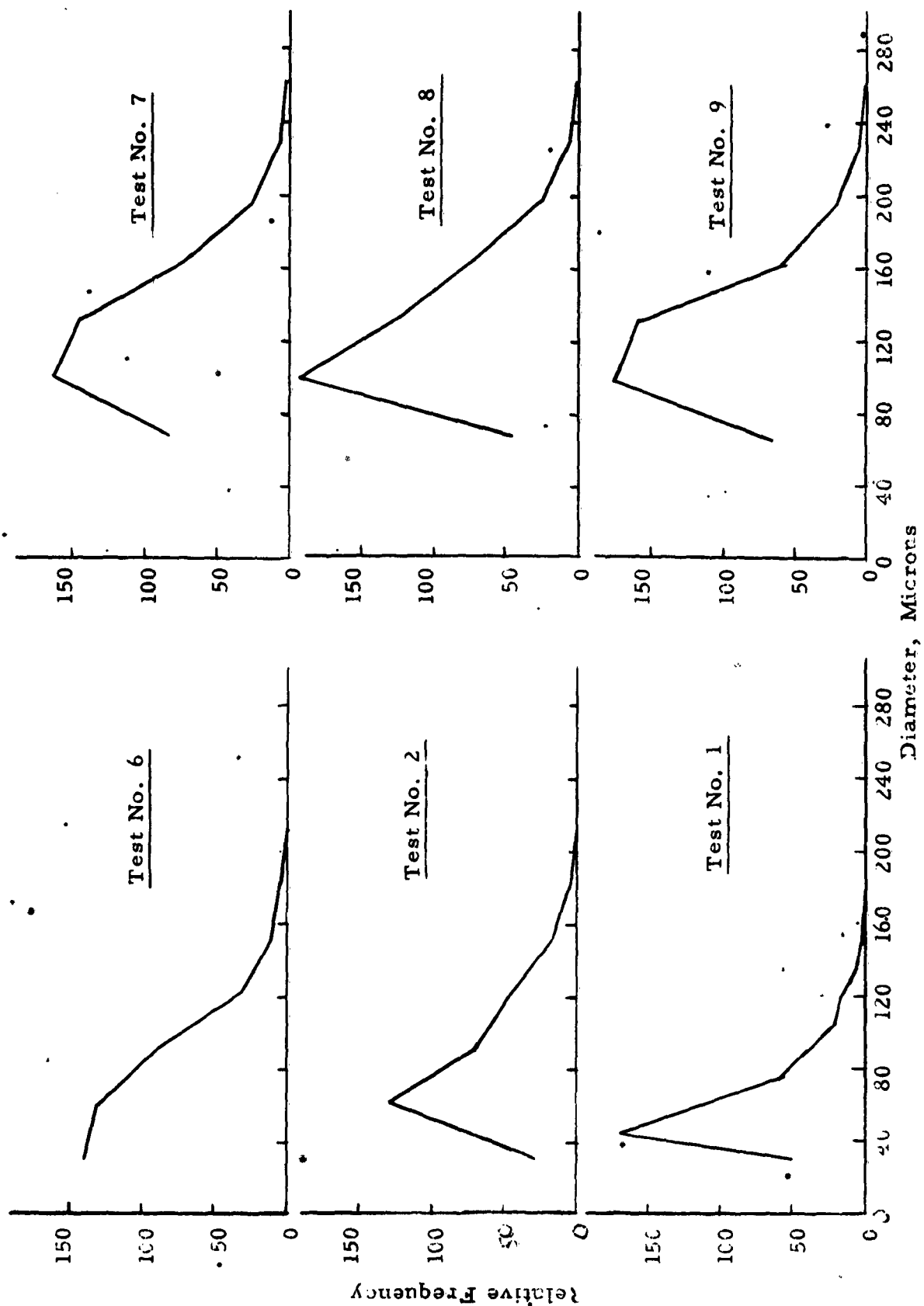


Fig. 5 TYPICAL DBP DROP SIZE FREQUENCY VARIATIONS

Table I
TEST DATA FOR DBP AEROSOLS

Test No.	Lean Limit, mg/l	Diameters, microns				DBP cc/min	Nitrogen l/min	Elutriating Air Velocity, cm/sec
		Arithmetic Mean ¹	Weight Mean ²	Sauter Mean ³	Weight Mean ⁴			
1	26.7	62	74	87	109	1.9	1.45	1.91
2	27.8	82	97	114	130	1.9	1.45	1.91
3	27.1	99	119	147	181	1.9	1.45	1.91
4	24.9	95	107	121	133	2.8	1.60	4.03
5	22.9	66	85	106	126	2.8	1.60	4.03
6	21.2	66	83	103	121	2.8	1.60	4.03
7	17.3	118	130	143	156	3.8	1.60	9.61
8	18.5	119	129	140	150	3.9	1.60	9.61
9	18.2	117	128	140	153	3.8	1.60	9.61

$$1 \quad \frac{1}{n} \sum d_i f_i$$

$$4 \quad \frac{\sum f_i d_i^4}{\sum f_i d_i^3}$$

$$2 \quad \sqrt[3]{\frac{\sum f_i d_i^3}{n}}$$

$$5 \quad \sum_0^m f_i d_i = 0.5 \sum_0^n f_i d_i$$

$$3 \quad \frac{\sum f_i d_i^3}{\sum f_i d_i^2}$$

m = size index of mass median drop
n = total number of size increments

The Sauter Mean Diameter ranges from 10 - to 25 per cent higher than the mean weight diameter, and, in general, the mean diameters tend to increase with an increase in either the fuel flow rate or the elutriating air velocity. Appreciable elutriation occurred only in tests 7, 8, and 9, and as a result these tests produced more nearly uniform aerosols.

V. FLAMMABILITY LIMITS FOR DBP

To make a limit determination, the generator is set to give an aerosol of the desired size and the rising air velocity is increased until the lean limit is approached. Minute adjustment of the fuel flow then permits attainment of the lean limit by successive trials. Then, after the coated slide samples (at least two per test) have been obtained, a 3-inch diameter millipore filter is placed at the bottom of the combustion tube simultaneously with the closing of the remote-operated valve above the tube. The aerosol contained in the tube settles and is collected by the filter and weighed. In the tests to date, no air flow through the filter has been necessary for efficient sampling, due to the high settling velocities of these coarse-sized aerosols. In fact, the aerosol invariably leaves a pattern on the millipore filter which is smaller in diameter, than the combustion tube, indicating that the diameter of the aerosol column is slightly smaller than the inside diameter of the combustion tube. The diameter of the aerosol column thus determined is used to calculate the mass concentration. At least two mass determinations are made for each test.

As noted in Table I and Fig. 6, there appears to be a definite trend toward a lower lean limit as the arithmetic mean drop diameter increases from 62 - to 119 microns. The corresponding lean limit, reported by Burgoyne and Cohen (Ref. 3) in their work on the lean limit of tetralin vs

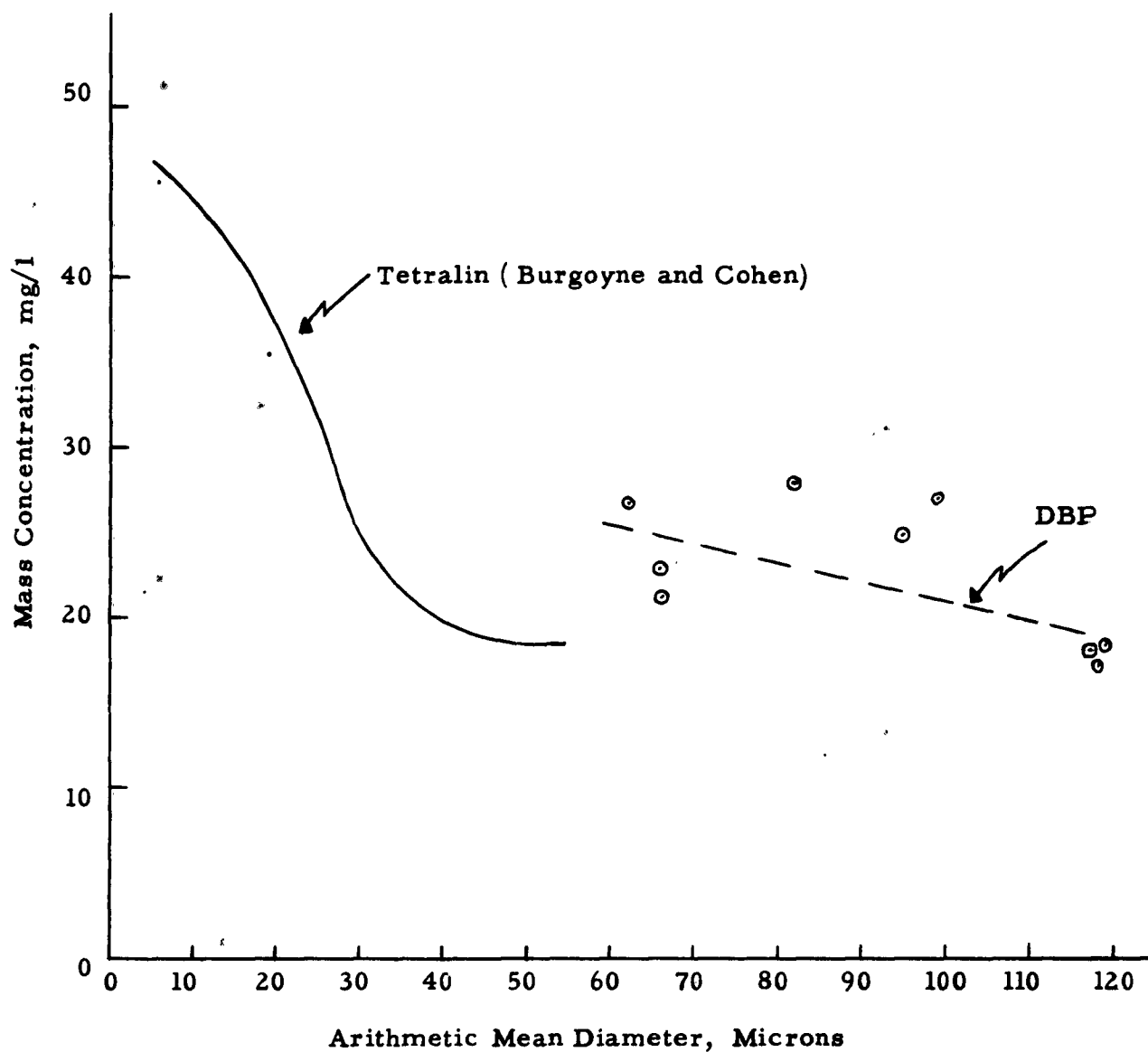


Fig. 6 VARIATION OF LEAN LIMIT WITH DROP SIZE

arithmetic mean diameter, is also shown. The curve for DBP appears to be displaced toward the larger drop sizes when compared with the curve for tetralin, a more volatile liquid, although the lowest lean limit for DBP coincides almost exactly with the lowest limit observed by Burgoyne and Cohen. A better understanding of the variation of the lean limit of DBP with drop size will be possible when additional data extending the range of mean drop sizes are available.

VI. PRELIMINARY TESTS WITH BIS (2-ETHYLHEXYL) HYDROGEN PHOSPHITE

Some qualitative tests were made with B2EHHP to determine the suitability of the thermal aerosol generation system for handling this chemical. When the test was started, the B2EHHP initially appeared to generate an aerosol in much the same manner as had the DBP. Upon ignition, the aerosol burned with a pinkish-yellow flame which produced a dense white smoke of oxidized phosphorous. After the first ignition, it was not possible to obtain a large drop aerosol, apparently due to the self-nucleating effect of the combustion products which must have contaminated the generator. Ignition of the subsequently produced self-nucleated B2EHHP aerosols of the order of 10 microns in size was easily accomplished, and combustion was evidenced by a strong flame which, on one occasion, propagated almost explosively through the length of the combustion tube. These experiments showed conclusively that B2EHHP aerosols can indeed support combustion. Unfortunately, the self-nucleating nature of this simulant makes its use in the thermal aerosol generator impractical for generation of the coarse-sized aerosols. This difficulty can be obviated through the use of a direct spray-type aerosol generator. By operating the large drop aerosol generator at high

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liquid pressures, drops in the size range of the thermally generated aerosols can be produced.

VII. SPONSOR VISITS

During the fifth quarter, the following CWL personnel visited the Foundation for a discussion of current project activity:

26 May 1961

Col. D. E. Yanka, Director of Research
and Development

12 July 1961

J. V. Pistritto, Project Monitor

Both Col. Yanka and Mr. Pistritto were favorably impressed with the demonstration of the experimental apparatus and expressed approval of future plans to investigate the effectiveness of both gaseous and liquid inhibitors. It was suggested that flame propagation rates be determined and that future investigations include the flammability of several similar liquids.

VIII. FUTURE WORK

Additional tests with DBP will be made to better define the lean limit curve, after which the inhibition tests will be started with gaseous inhibitors in DBP aerosols. Construction of the large drop aerosol generator will be completed for use in the generation of large drop DBP aerosols, and both large and small drop B2EHHP aerosols. Liquid and solid type inhibitors will be evaluated with the large drop aerosol generator. Tetralin will also be used in the thermal generator, for comparison with the data of Burgoyne and Cohen (Ref. 3), and to extend their range of drop sizes. A phosphate type simulant more nearly related to the agents in question may be used in the final flammability and inhibition study, to verify the effectiveness of any inhibitors found promising with DBP and B2EHHP. The Sponsor will be notified immediately of any promising inhibitors, so that the compatibility

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of the inhibitors with the agent may be determined. Physical properties of the various simulants will be determined, so that the theory presented in the Fourth Quarterly Progress Report can be applied directly to the conditions of the experimental tests.

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2. May, K. R., J. Sci. Instr., 27, 128 (1950)
3. Burgoyne, J. H., and Cohen, L., Proc. Roy. Soc., 225A, 375 (1954)

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